

Substantial Changes in the Characteristics of a Microwave Plasma Due to Combining Argon and Hydrogen

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ABSTRACT

Upon the addition of 5% argon to a hydrogen plasma, the Lyman α emission was observed to increase by about an order of magnitude; whereas, xenon control had no effect. With a microwave input power of 40 W, the gas temperature of an argon plasma increased from 400°C to over 750°C with the addition of 3% flowing hydrogen; whereas, the 400°C temperature of a xenon plasma run under identical conditions was essentially unchanged with the addition of hydrogen. The average hydrogen atom temperature of the argon-hydrogen plasma was measured to be 110 - 130 eV versus ≈ 3 eV for pure hydrogen or xenon-hydrogen. Stark broadening or acceleration of charged species due to high fields (e. g. over 10 kV/cm) can not be invoked to explain the results with argon since no high field was observationally present. The electron temperature T_e for the argon-hydrogen and xenon-hydrogen plasmas was $11,600 \pm 5\% K$ and $6500 \pm 5\% K$, respectively, compared to $4800 \pm 5\% K$ and $4980 \pm 5\% K$ for argon and xenon alone, respectively. The observation of higher temperatures corresponding to three possibly independent plasma parameters for only argon with hydrogen may be explained by the release of energy from atomic hydrogen by a resonant nonradiative energy transfer mechanism.

I. INTRODUCTION

It was reported previously that a new plasma source has been developed that operates by incandescently heating a hydrogen dissociator to provide atomic hydrogen and heats a catalyst such that it becomes gaseous and reacts with the atomic hydrogen to produce a plasma called a resonance transfer or rt-plasma. It was extraordinary, that intense VUV emission was observed by Mills et al. [1-2] at low temperatures (e.g. $\approx 10^3$ K) and an extraordinary low field strength of about 1-2 V/cm from atomic hydrogen and certain atomized elements or certain gaseous ions which singly or multiply ionize at integer multiples of the potential energy of atomic hydrogen, 27.2 eV.

Prior related studies that support the possibility of a novel reaction of atomic hydrogen which produces a chemically generated or assisted plasma (rt-plasma) and produces novel hydride compounds include EUV spectroscopy [1-12, 14], characteristic emission from catalysts and the hydride ion products [8-11], lower-energy hydrogen emission [3-5, 7], chemically formed plasmas [1-2, 6, 8-12], Balmer α line broadening [3-5, 11, 13, 15], elevated electron temperature [3, 5, 13], anomalous plasma afterglow duration [6, 14], power generation [2, 3, 12, 15, 16], and analysis of novel chemical compounds [17]. Argon ions can provide a net enthalpy of a multiple of that of the potential energy of the hydrogen atom. The second ionization energy of argon is 27.63 eV [18]. The reaction Ar^+ to Ar^{2+} has a net enthalpy of reaction of 27.63 eV. Thus, an argon microwave discharge with hydrogen present was anticipated to form an rt-plasma, and the effect of the addition of hydrogen to an argon microwave plasma compared to xenon control was determined. The plasmas were characterized by measuring plasma gas temperature, the ion temperature and number density from Balmer α line broadening, and the electron temperature T_e from intensity ratios of alkali lines.

II. EXPERIMENTAL

A. EUV spectroscopy of hydrogen microwave plasmas with the addition of 5% argon or xenon

Extreme ultraviolet (EUV) spectroscopy was recorded on a hydrogen microwave plasma alone and with the addition of 5% argon or 5% xenon. Due to the short wavelength of this radiation, "transparent" optics do not exist. Therefore, a windowless arrangement was used wherein the microwave discharge cell was connected to the same vacuum vessel as the grating and detectors of the EUV spectrometer. Differential pumping permitted a high pressure in the

cell as compared to that in the spectrometer. This was achieved by pumping on the cell outlet and pumping on the grating side of the collimator that served as a pin-hole inlet to the optics. The spectrometer was continuously evacuated to 10^{-4} – 10^{-6} Torr by a turbomolecular pump with the pressure read by a cold cathode pressure gauge. The EUV spectrometer was connected to the cell light source with a 1.5 mm X 5 mm collimator which provided a light path to the slits of the EUV spectrometer. The collimator also served as a flow constrictor of gas from the cell. The cell was operated under gas flow conditions while maintaining a constant gas pressure in the cell.

For spectral measurement, the light emission from microwave plasmas of hydrogen alone, hydrogen-argon (95/5%), and hydrogen-xenon (95/5%) were introduced to a normal incidence McPherson 0.2 meter monochromator (Model 302, Seya-Namioka type) equipped with a 1200 lines/mm holographic grating with a platinum coating. The wavelength region covered by the monochromator was 5 – 560 nm. The UV spectrum (90 – 165 nm) of the cell emission was recorded with a photomultiplier tube (PMT) and a sodium salicylate scintillator. The PMT (Model R1527P, Hamamatsu) used has a spectral response in the range of 185 – 680 nm with a peak efficiency at about 400 nm. The wavelength resolution was about 1 nm (FWHM) with an entrance and exit slit width of 300 μ m. The increment was 0.1 nm and the dwell time was 500 ms.

B. Microwave Emission Spectra

The experimental set up comprising a microwave discharge gas cell light source and an EUV spectrometer which was differentially pumped is shown in Figure 1. Extreme ultraviolet emission spectra were obtained on plasmas of hydrogen alone, hydrogen-argon mixture (95/5%), and hydrogen-xenon mixture (95/5%). Hydrogen or the hydrogen-noble gas mixture was flowed through a half inch diameter quartz tube at 1 Torr that was maintained by flowing hydrogen or the gas mixture while monitoring the pressure with a 10 Torr and 1000 Torr MKS Baratron absolute pressure gauge. The tube was fitted with an Ophos coaxial microwave cavity (Evenson cavity). The microwave generator shown in Figure 1 was an Ophos model MPG-4M generator (Frequency: 2450 MHz). The input power to the plasma was set at 85 watts. The EUV spectrometer was a normal incidence monochromator. (See Section II A).

C. Gas temperature measurements on microwave discharge plasmas

In order to estimate the relative power output [19], the gas temperature of microwave plasmas of argon and xenon alone and each noble gas with 10% hydrogen was measured. The

experimental set up is described in Section II B. Each ultra-pure test gas or mixture was flowed through the half inch diameter quartz tube at 0.3 Torr maintained with a noble gas flow rate of 9.3 sccm or a noble gas flow rate of 8.3 sccm and a hydrogen flow rate of 1 sccm. The light emission was introduced into the normal incidence EUV spectrometer to determine the electron temperature as discussed in Section II E. Balmer α emission from the cell was also recorded with a high resolution visible spectrometer to determine the ion energy and density as discussed in Section II D.

With the input power to the plasma set at 40 watts, the temperature rise and fall was recorded using a K-type thermocouple ($\pm 0.1^\circ\text{C}$) housed in a stainless steel tube that was placed axially inside the center of the 10 cm^3 plasma volume of a quartz microwave cell as hydrogen flow supplied by the mass flow controller was turned on and off. The cell was operated under flow conditions with continuous pumping by the turbopump of the EUV spectrometer.

D. Balmer α line broadening recorded on microwave discharge plasmas

The method of Videnovic et al. [20] was used to calculate the energetic hydrogen atom densities and energies from the width of the 656.3 nm Balmer α line emitted from glow discharge and microwave plasmas. The full half-width $\Delta\lambda_G$ of each Gaussian results from the Doppler ($\Delta\lambda_D$) and instrumental ($\Delta\lambda_I$) half-widths:

$$\Delta\lambda_G = \sqrt{\Delta\lambda_D^2 + \Delta\lambda_I^2} \quad (4)$$

$\Delta\lambda_I$ in our experiments was 0.006 nm . The temperature was calculated from the Doppler half-width using the formula:

$$\Delta\lambda_D = 7.16 \times 10^{-7} \lambda_0 \left(\frac{T}{\mu} \right)^{1/2} \quad (\text{nm}) \quad (5)$$

where λ_0 is the line wavelength in nm, T is the temperature in K ($1\text{ eV} = 11,605\text{ K}$), and μ is the molecular weight ($=1$ for hydrogen). In each case, the average Doppler half-width that was not appreciably changed with pressure varied by $\pm 5\%$ corresponding to an error in the energy of $\pm 5\%$. The corresponding number densities for noble gas-hydrogen mixtures varied by $\pm 20\%$ depending on the pressure.

The width of the 656.3 nm Balmer α line was measured on light emitted from microwave discharges of pure hydrogen alone and a mixture of 10% hydrogen and argon or xenon. The experimental set is described in Section II C. The plasma emission was fiber-optically coupled through a 220F matching fiber adapter positioned 2 cm from the cell wall to a high resolution visible spectrometer with a resolution of $\pm 0.006\text{ nm}$ over the spectral range 190 - 860 nm. The spectrometer was a Jobin Yvon Horiba 1250 M with 2400 groves/mm ion-

etched holographic diffraction grating. The entrance and exit slits were set to $20 \mu m$. The spectrometer was scanned between $655.5 - 657 \text{ nm}$ using a 0.005 nm step size. The signal was recorded by a PMT with a stand alone high voltage power supply (950 V) and an acquisition controller. The data was obtained in a single accumulation with a 1 second integration time.

E. T_e measurements of microwave discharge plasmas

The most commonly used spectroscopic diagnostic method to determine the electron temperature T_e of laboratory plasmas is based on determining the relative intensities of two spectral lines as described by Griem [21]. It may be shown that for two emission lines at wavelengths λ_A and λ_B

$$\frac{I_A}{I_B} = \frac{(\sigma g_2 A_{21})_A}{(\sigma g_2 A_{21})_B} e^{-\frac{(E_{2A} - E_{2B})}{kT_e}} \quad (6)$$

where I_A and I_B are the intensities measured at λ_A and λ_B , and $\sigma \propto n^4$ for atomic hydrogen. The frequency ν , the transition probability A , the degeneracy g , and the upper level E are known constants from which T_e was determined.

T_e was measured on microwave plasmas of argon alone and argon-hydrogen mixture (90/10%) from the ratio of the intensity of the Ar 104.8 nm (upper quantum level $n = 3$) line to that of the Ar 420.06 nm ($n = 4$) line. T_e was also measured by the same method on microwave plasmas of pure hydrogen alone, xenon alone, and a mixture of 10% hydrogen and xenon using the ratio of the intensities of two hydrogen or xenon lines in two quantum states.

The experimental set up comprising a microwave discharge gas cell light source and an EUV spectrometer which was differentially pumped is shown in Figure 1. In each case, the microwave plasma cell was run under the conditions given in Section II C. The EUV-UV-VIS spectrum ($20 - 560 \text{ nm}$) of the cell emission was recorded with the Spectrometer described in Section II A. The spectra were repeated five times per experiment and were found to be reproducible within less than $\pm 5\%$.

III. RESULTS AND DISCUSSION

A. Argon-hydrogen microwave emission spectrum

The EUV spectrum ($90 - 165 \text{ nm}$) of the cell emission from the hydrogen plasma (dotted line) and the hydrogen plasma to which 5% argon was added (solid line) is shown in Figure 2. Upon the addition of 5% argon, the hydrogen Lyman α emission intensity was

observed to increase by about an order of magnitude. Essentially no effect was observed for the addition of 5% xenon to the hydrogen plasma. This result indicates that one or more temperatures may be elevated with the addition of argon to a hydrogen plasma.

B. Gas temperature measurements

Due to the high mobility of free electrons, the heat loss of the microwave cell was determined by slow losses from the cell walls with a fast power transfer from the plasma to the wall such that the plasma was isothermal and the inner wall temperature and the plasma gas temperatures were equivalent as discussed by Chen et al. [19]. Since the microwave discharge cell, power input to the plasma, and discharge conditions remained identical between the argon and xenon experiments, the gas temperature may be used to determine the power balance as shown by Chen et al [19].

Essentially no increase in gas temperature was observed with the addition of hydrogen to xenon control as shown in Figure 3. In contrast, With a microwave input power of 40 *W*, the gas temperature of an argon plasma increased from 400°C to over 750°C with the addition of 3% flowing hydrogen as shown in Figure 4. A conservative estimate of the total output power was determined by taking the ratio of the maximum ΔT , the cell temperature increase above the ambient temperature of $25.0 \pm 0.1^\circ\text{C}$, of the argon-hydrogen plasma compared to that of the argon alone, xenon alone, and xenon-hydrogen plasmas, 1.9, multiplied by the common input. Thus, with a microwave input power of 40 *W*, the thermal output power was estimated to be 76 *W*. Since the hydrogen flow rate was 1 sccm, an estimate of the corresponding energy balance was over $-1 \times 10^4 \text{ kJ/mole } H_2$ compared to the enthalpy of combustion of hydrogen of $-241.8 \text{ kJ/mole } H_2$.

C. Line broadening and T_e measurements

The 656.3 nm Balmer α line width recorded with a high resolution ($\pm 0.006 \text{ nm}$) visible spectrometer on microwave discharge plasmas of hydrogen compared with each of xenon-hydrogen (90/10%) and argon-hydrogen (90/10%) are shown in Figures 5 and 6, respectively. The energetic hydrogen atom densities and energies of plasmas of hydrogen alone and the noble gas-hydrogen mixtures were calculated using the method of Videnovic et al. [20]. It was found that the microwave argon-hydrogen plasma showed extraordinary broadening corresponding to an average hydrogen atom temperature of 110-130 *eV* and an atom density of $3.5 \times 10^{14} \text{ atoms/cm}^3$. Whereas, xenon-hydrogen and pure hydrogen showed no excessive broadening corresponding to an average hydrogen atom temperature of 3-4 *eV* for both and

an atom density of only $3 \times 10^{13} \text{ atoms/cm}^3$ and $7 \times 10^{13} \text{ atoms/cm}^3$, respectively, even though 10 times more hydrogen was present for pure hydrogen.

These studies demonstrate excessive line broadening in the absence of an observable effect attributable to an electric field since the hydrogen emission shows no broadening. Since no electric field was present in the microwave plasma, the results can not be explained by Stark broadening or acceleration of charged species due to high fields of over 10 kV/cm as proposed by Videnovic et al. [20] to explain excessive broadening observed in glow discharges. Excessive line broadening was only observed in the cases where Ar^+ was present which could provide a net enthalpy of reaction of an integer multiple of the potential energy of atomic hydrogen. Whereas, plasmas of chemically similar xenon control that do not provide gaseous atoms or ions that have electron ionization energies which are a multiple of 27.2 eV showed no effect with the addition of 10% hydrogen. These results support the rt-plasma mechanism.

Rt-plasmas formed with hydrogen-potassium mixtures have been reported previously [6, 14] wherein the plasma decayed with a two second half-life when the electric field was set to zero. This was the thermal decay time of the filament which dissociated molecular hydrogen to atomic hydrogen. This experiment showed that hydrogen line emission was occurring even though the voltage between the heater wires was set to and measured to be zero, and it indicated that the emission was due to a reaction of potassium atoms with atomic hydrogen. Potassium atoms ionize at an integer multiple of the potential energy of atomic hydrogen, $m \cdot 27.2 \text{ eV}$. The enthalpy of ionization of K to K^{3+} has a net enthalpy of reaction of 81.7426 eV , which is equivalent to $m = 3$. K^{3+} and the formation of the corresponding hydride were detected by EUV spectroscopy recorded on an rt-plasma [9].

Similarly, to the ion measurement, the average electron temperature T_e for the argon-hydrogen plasma was high, $11,600 \pm 5\% \text{ K}$, compared to $4800 \pm 5\% \text{ K}$, $4980 \pm 5\% \text{ K}$, and $6500 \pm 5\% \text{ K}$ for argon alone, xenon alone, and xenon-hydrogen plasmas, respectively.

IV. CONCLUSION

Upon the addition of 5% argon to a hydrogen plasma, the Lyman alpha emission was observed to increase by about an order of magnitude which suggested that one or more of the plasma temperatures may be elevated; whereas, no effect was observed with xenon. Line broadening of the hydrogen Balmer lines provides a sensitive measure of the number and energy of excited hydrogen atoms in a plasma. The width of the 656.3 nm Balmer α line emitted from microwave discharge plasmas having atomized hydrogen from pure hydrogen alone and a mixture of 10% hydrogen and argon or xenon was measured with a high resolution ($\pm 0.006 \text{ nm}$) visible spectrometer. The energetic hydrogen atom density and energies were

determined from the broadening, and it was found that argon-hydrogen showed significant broadening corresponding to an average hydrogen atom temperature of 110 - 130 eV; whereas, pure hydrogen and xenon-hydrogen showed no excessive broadening corresponding to an average hydrogen atom temperature of ≈ 3 eV. Similarly, the average electron temperature T_e for the argon-hydrogen was high, $11,600 \pm 5\% K$, compared to $4800 \pm 5\% K$, $4980 \pm 5\% K$, and $6500 \pm 5\% K$ for argon alone, xenon alone, and xenon-hydrogen plasmas, respectively.

The gas temperature is a means to estimate the power output of the cell. With a microwave input power of 40 W, the gas temperature of an argon plasma increased from 400°C to over 750°C with the addition of 3% flowing hydrogen; whereas, the 400°C temperature of a xenon plasma run under identical conditions was essentially unchanged with the addition of hydrogen. The thermal output power was estimated to be 76 W. Since the hydrogen flow rate was 1 sccm, an estimate of the corresponding energy balance was over -1×10^4 kJ/mole H_2 compared to the enthalpy of combustion of hydrogen of -241.8 kJ/mole H_2 .

The observed excessive line broadening, elevated T_e , and elevated plasma gas temperature were only observed for the case were Ar^+ , an ion which provides a net enthalpy of reaction of a multiple of the potential energy of the hydrogen atom, was present with atomic hydrogen. Nonequilibrium plasma conditions may explain the elevation of one temperature over another [22]; however, the elevation of all three temperatures indicates power dissipation in the plasma in addition to the microwave input. The source of additional power corresponding to the elevated temperatures may be an energetic reaction of atomic hydrogen caused by a resonance energy transfer between hydrogen atoms and Ar^+ .

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Figure Captions

Figure 1. The experimental set up comprising a microwave discharge gas cell light source and an EUV-UV-VIS spectrometer which was differentially pumped.

Figure 2. The EUV spectrum (90 – 165 nm) of the cell emission from the hydrogen plasma (dotted line) and the hydrogen plasma to which 5% argon was added (solid line).

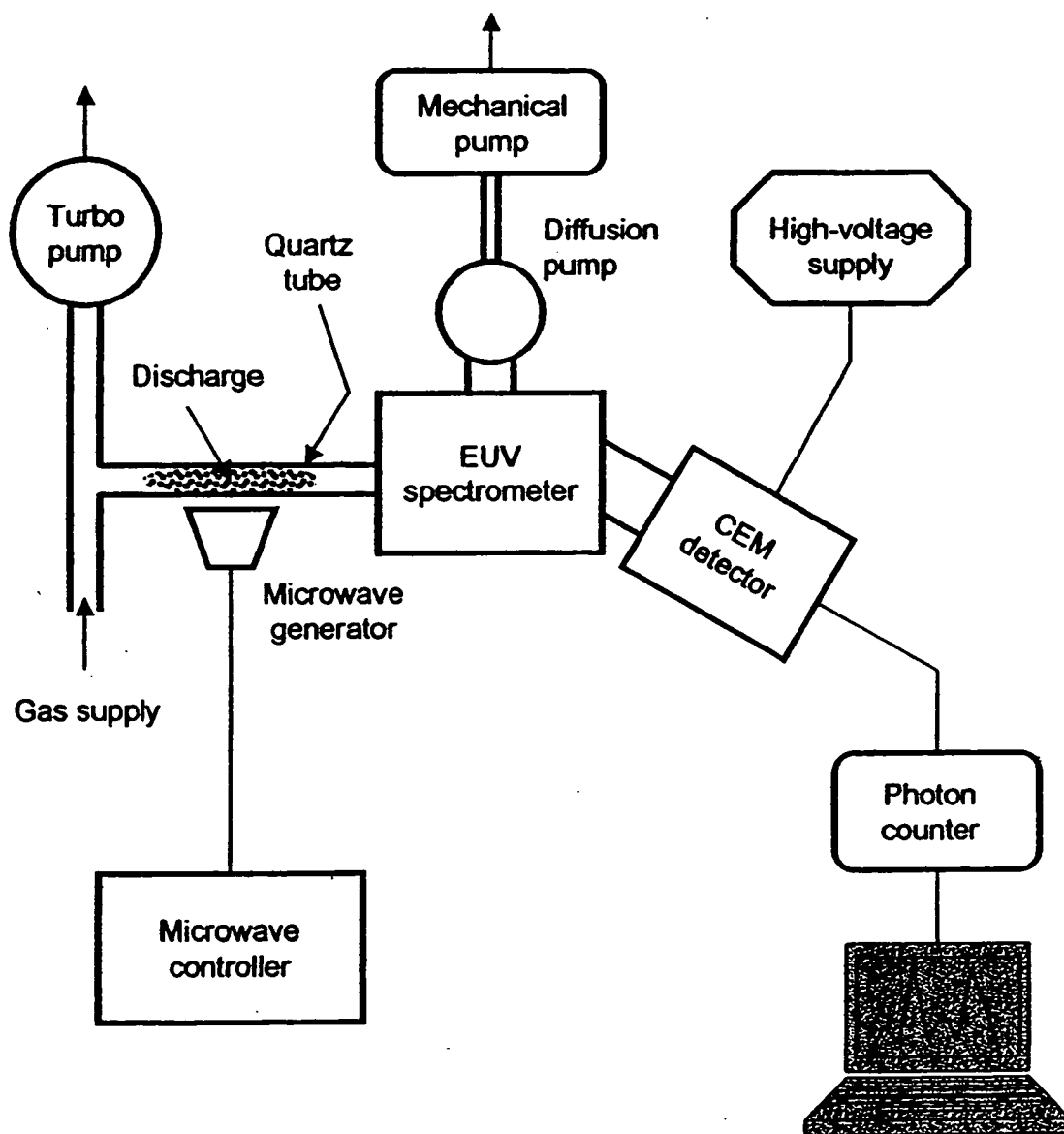
Figure 3. The plasma gas temperature rise as a function of time for xenon alone and the xenon-hydrogen mixture (90/10%) with the microwave input power set at 40 W as the hydrogen flow was turned on and off. The 400°C plasma gas temperature was essentially unchanged with the addition of hydrogen.

Figure 4. The plasma gas temperature rise as a function of time for argon alone and the argon-hydrogen mixture (90/10%) with the microwave input power set at 40 W as the hydrogen flow was turned on and off. The plasma gas temperature reproducibly increased from 400°C to over 750°C with the addition of 3% flowing hydrogen. The thermal output power of the argon-hydrogen plasma was estimated to be 76 W.

Figure 5. The 656.3 nm Balmer α line width recorded with a high resolution (± 0.006 nm) visible spectrometer on a xenon-hydrogen (90/10%) and a hydrogen microwave discharge plasma. No line excessive broadening was observed corresponding to an average hydrogen atom temperature of 3 – 4 eV.

Figure 6. The 656.3 nm Balmer α line width recorded with a high resolution (± 0.006 nm) visible spectrometer on a argon-hydrogen (90/10%) and a hydrogen microwave discharge plasma. Significant broadening was observed corresponding to an average hydrogen atom temperature of 110 - 130 eV.

Fig. 1



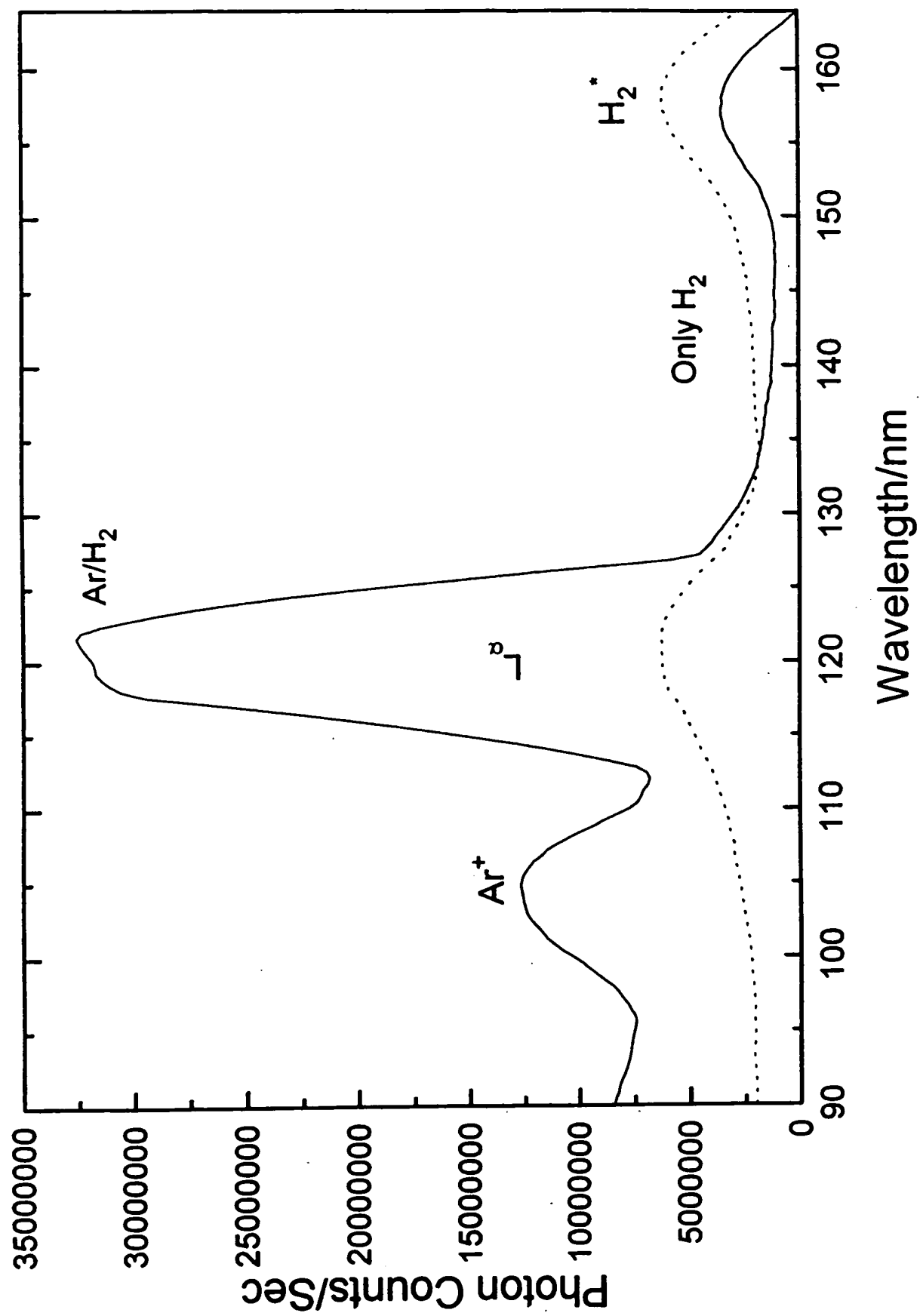


Fig. 2

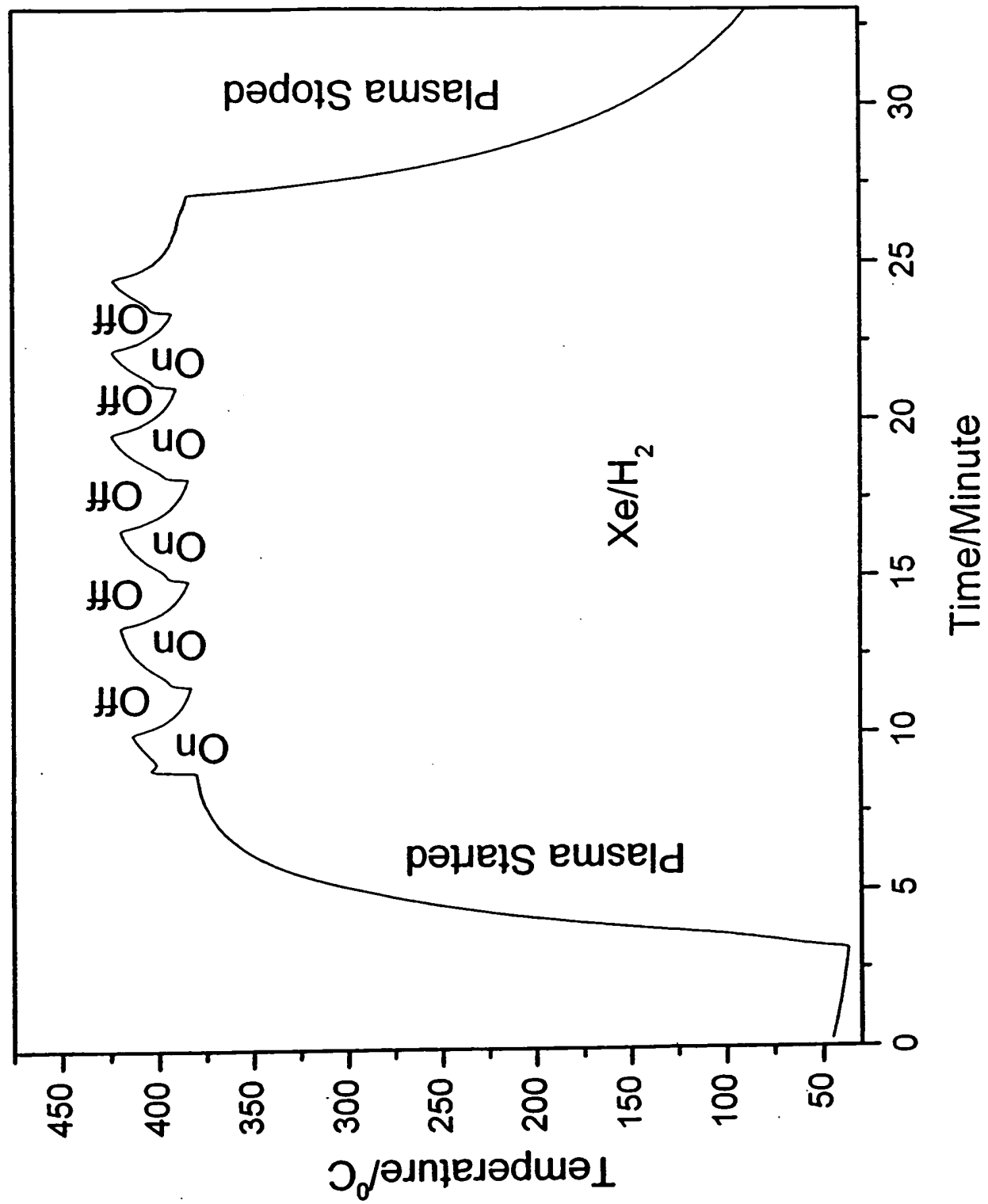


Fig. 3

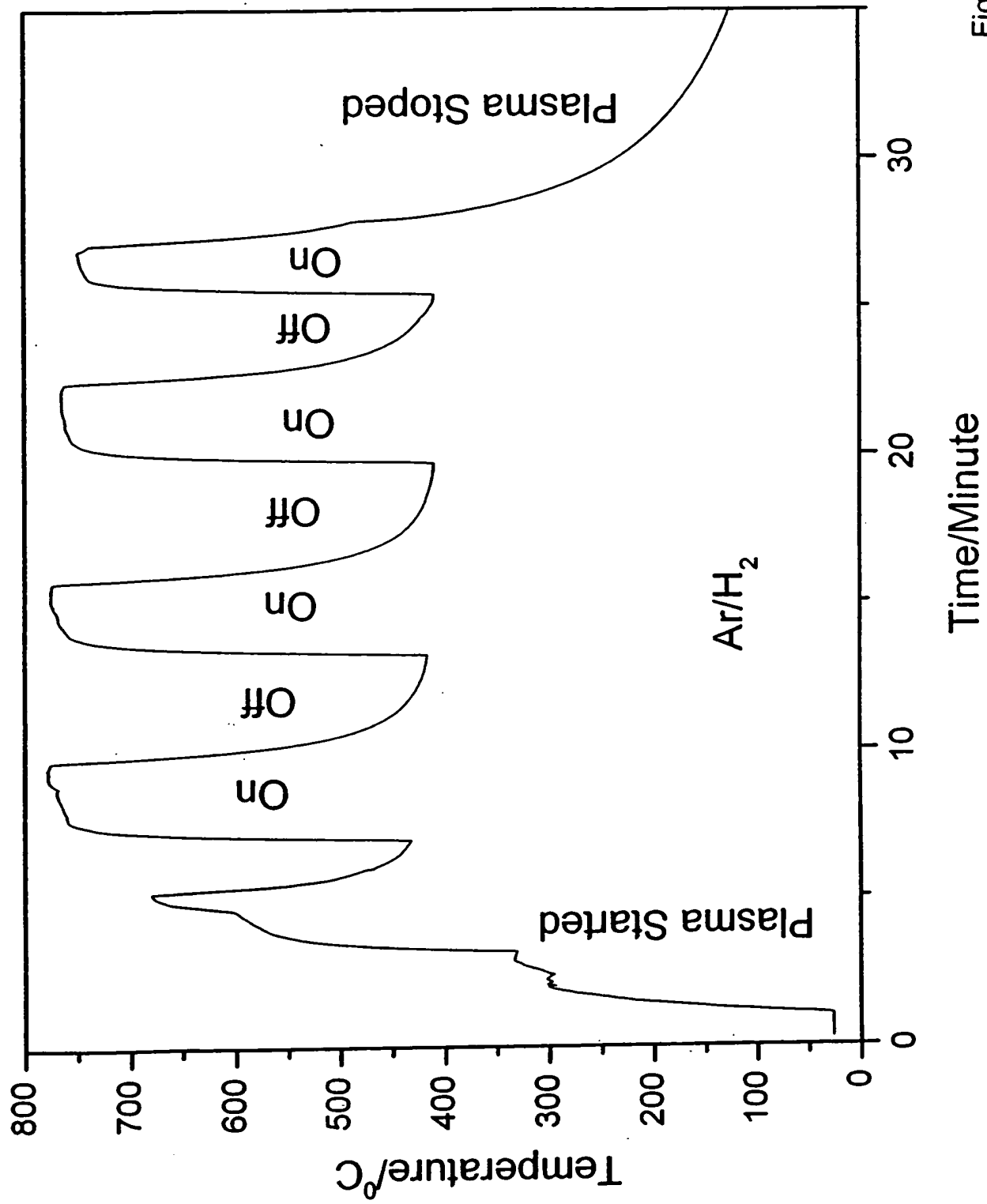


Fig. 4

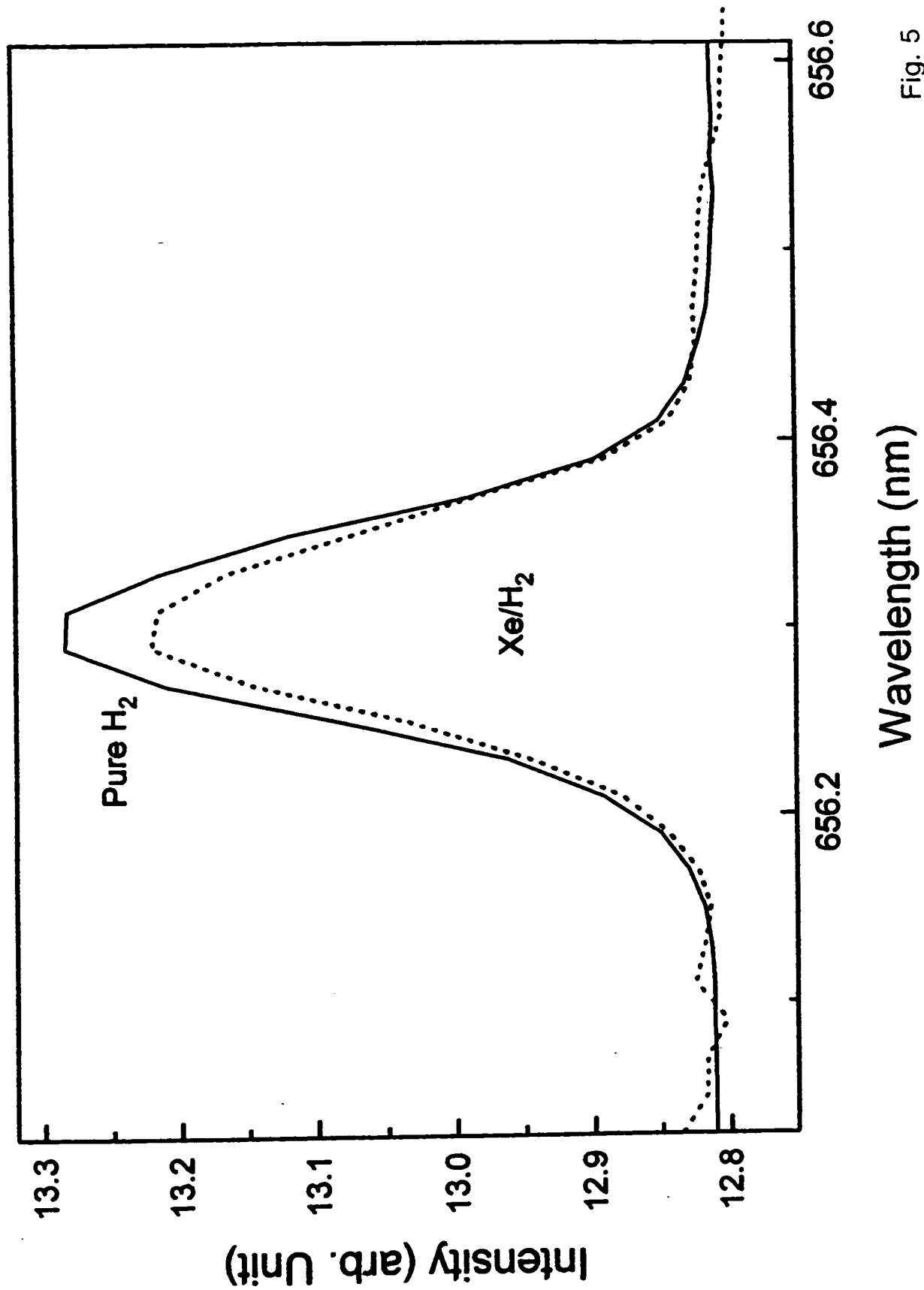


Fig. 5

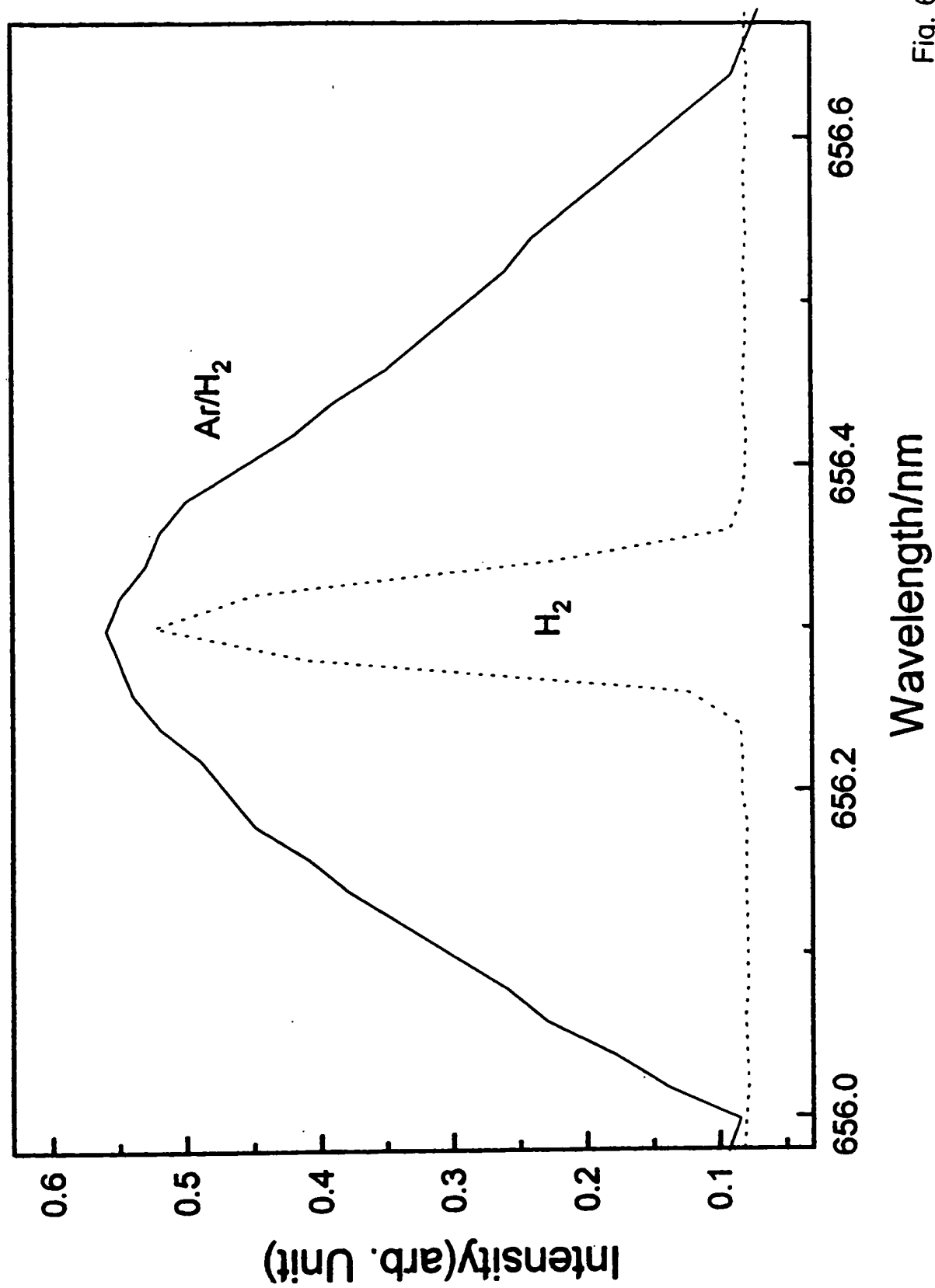


Fig. 6

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